Claims

1. Apparatus for parallel processing of multiple reaction mixtures, said apparatus comprising

a reactor having an exterior surface,

vessels in the reactor for holding said reaction mixtures, each vessel having a central longitudinal axis,

a cannula for introducing fluid reaction material into the vessels, said cannula having a longitudinal axis, a distal end, and a port generally adjacent said distal end for delivery of said reaction material from the cannula,

cannula passages in the reactor extending between said exterior surface of the reactor and said vessels, each cannula passage extending at an angle relative to said central longitudinal axis of a respective vessel, and

a robot system operable to insert the cannula through a selected cannula passage and into a respective vessel for the delivery of said reaction material from the cannula to the respective vessel, and to withdraw the cannula from the selected cannula passage and respective vessel.

2. Apparatus as set forth in claim 1 wherein said port of the cannula opens generally laterally with respect to the longitudinal axis of the cannula whereby reaction material is delivered from the cannula and directed into said vessels in a downward direction generally parallel to said central longitudinal axis of each vessel.

3. Apparatus as set forth in claim 1 further comprising a sealing mechanism in each cannula passage for sealing

against leakage of gas therepast when the cannula is inserted into and withdrawn from the cannula passages.

4. Apparatus as set forth in claim 3 wherein said sealing mechanism comprises a valve movable between a closed position for closing the cannula passage and an open position permitting movement of the cannula through the passage, and a seal in the passage sealingly engageable with the cannula when the valve is in its open position.

5. Apparatus as set forth in claim 3 wherein said seal is located on a side of the valve opposite the vessel.

6. Apparatus as set forth in claim 1 further comprising a wiper on the reactor adjacent an inlet end of each cannula passage for wiping the exterior surface of the cannula as it is inserted in the passage to wipe away any material thereon.

7. Apparatus as set forth in claim 1 wherein said robot system is operable to move said cannula to an angled orientation in which the cannula is held at an angle corresponding to the angle the selected cannula passage extends relative to said central longitudinal axis of the respective vessel for insertion of the cannula into the selected cannula passage.

8. Apparatus as set forth in claim 7 further comprising a sealing mechanism in each cannula passage for sealing against leakage of gas therepast when the cannula is inserted into and withdrawn from the cannula passages, said sealing mechanism comprising a valve movable between a closed position for closing the passage and an open position permitting movement of the cannula through the passage, and a seal in the passage sealingly engageable with the cannula

when the valve is in its open position, said seal being located on a side of the valve opposite said vessel.

9. Apparatus as set forth in claim 8 wherein said robot system is operable to insert said cannula in said angled orientation into a cannula passage to a delivery position in which the distal end of the cannula is downstream from the valve for delivery of reaction material to a respective vessel, then to withdraw the cannula to an intermediate position in which the distal end of the cannula is between the valve and said seal, the robot system holding the cannula in said intermediate position for a dwell period sufficient to allow the valve to close prior to completely withdrawing the cannula from the cannula passage.

10. Apparatus as set forth in claim 7 wherein said robot system comprises an arm rotatable about a longitudinal axis extending generally parallel to the arm, a mount on the arm for mounting said cannula, and a rotating mechanism for rotating the arm about said longitudinal axis to move the cannula between said angled position and a generally vertical position.

11. Apparatus as set forth in claim 10 wherein said rotating mechanism comprises an actuator for rotating said arm in two directions, a first stop for limiting rotation of the arm in one direction to stop the arm at a position corresponding to said angled position of the cannula, and a second stop for limiting rotation of the arm in an opposite direction to stop the arm at a position corresponding to said generally vertical position of the cannula.

12. Apparatus as set forth in claim 11 wherein said actuator comprises a double-acting power cylinder.

13. Apparatus as set forth in claim 10 wherein said cannula comprises a long metal tube, and wherein said apparatus further comprises a cannula support on the mount engageable with the tube intermediate the ends of the tube for supporting and stabilizing the tube in precise position as the cannula is moved.

14. Apparatus as set forth in claim 13 wherein said cannula support comprises a body affixed to the cannula mount and a head mounted on the body and having an opening therein sized for a close clearance fit with said long metal tube, said head being movable relative to the body from an extended position in which the head is spaced from the body for engagement with a more distal portion of the tube, and a retracted position in which the head is closer to the body for engagement with a more proximal portion of the tube to allow for insertion of the said more distal portion of the tube into a cannula passage.

15. Apparatus as set forth in claim 1 wherein said cannula comprises a reservoir for holding a volume of said reaction material, said reservoir having an outside diameter, and a long thin tubular needle in fluid communication with said reservoir and having an outside diameter less than the outside diameter of the reservoir, said needle having a lateral opening constituting said port of the cannula.

16. Apparatus as set forth in claim 1 further comprising a heated wash tower having a well therein for receiving a portion of a cannula to be cleaned, said tower having a heater for heating fluid in the well to clean said portion of the cannula.

17. A method of loading fluid reaction material into a series of vessels in a reactor, each vessel having a central longitudinal axis, said method comprising, in sequence:

(1) inserting a cannula through a cannula passage in said reactor to a position in which the cannula extends at an angle relative to the central longitudinal axis of a first vessel of said series of vessels, and in which a distal end of the cannula is disposed in said vessel,

(2) delivering a fluid reaction material from said cannula into the vessel,

(3) withdrawing the cannula from said passage, and

(4) repeating 1-3 for a second vessel.

18. A method as set forth in claim 17 wherein the cannula has a port adjacent its said distal end opening laterally relative to a longitudinal axis of the cannula, said method further comprising orienting said distal end of the cannula in the vessel so that said port faces downwardly for delivering reaction material from the cannula in a downward direction.

19. A method as set forth in claim 17 wherein said cannula has an outside surface, and wherein said method further comprises wiping the outside surface as the cannula is inserted in said cannula passage.

20. A method as set forth in claim 17 wherein each cannula passage has a sealing mechanism therein for sealing against the leakage of gas therepast when the cannula is inserted into and withdrawn from the passages, said method comprising inserting the cannula into said cannula passage to a point past said sealing mechanism, and then delivering

pressurized fluid reaction material from the cannula into the vessel.

21. A method as set forth in claim 20 wherein said sealing mechanism comprises a valve movable between a closed position for closing the cannula passage and an open position permitting movement of the cannula through the passage, and a seal in the passage sealingly engageable with the cannula when the valve is in its open position, said seal being located on a side of the valve opposite said vessel, said withdrawing step comprising withdrawing the cannula to an intermediate position in which the distal end of the cannula is located between the valve and said seal, and holding the cannula in said intermediate position for a dwell period sufficient to allow the valve to close before completely withdrawing the cannula from the cannula passage.

22. A method as set forth in claim 17 wherein said reaction material is a slurry comprising a catalyst fluid.

23. A method as set forth in claim 21 wherein said catalyst is disposed on a particulate support.

24. A method as set forth in claim 17 further comprising cleaning the cannula after withdrawing it from said cannula passage, said cleaning comprising washing and rinsing the cannula using a heated solution.

25. A method as set forth in claim 17 further comprising cleaning the cannula after withdrawing it from said cannula passage, said cleaning comprising subjecting said cannula to ultrasonic waves.

26. A cannula for use aspirating reactant materials and delivering such materials to reaction vessels for the parallel processing of such materials, said cannula

comprising

a tubular metal reservoir having a longitudinal axis, an inside diameter defining a hollow interior for containing said reactant materials, an outside diameter, a proximal end and a distal end,

a long straight thin needle formed from metal tubing and coaxial with said reservoir, said needle having an outside diameter substantially less than the outside diameter of the reservoir and an inside diameter defining a flow passage through the needle, said needle further having a proximal end, a distal end, and a port adjacent said distal end for aspirating said reactant materials into the needle and delivering reactant materials from the needle,

a metal transition joining the proximal end of the needle to the distal end of said reservoir so that the hollow of the interior of the reservoir is in fluid communication with the flow passage of the needle.

27. A cannula as set forth in claim 26 wherein the reservoir, needle and transition have interior surfaces which are seamed together to form a continuous interior expanse of smooth metal extending from the reservoir to the transition to the needle.

28. A cannula as set forth in claim 27 wherein said transition is generally funnel-shaped to have an sloping side wall, a bottom wall, and a hole through the bottom wall receiving a distal end portion of the needle, the distal end of the needle being flush with the interior surface of the transition.

29. A cannula as set forth in claim 28 wherein said transition is joined to the reservoir and needle by welds, and wherein the welds are polished to a smooth finish on the inside of the cannula.

30. A cannula as set forth in claim 26 wherein the port of the needle faces laterally away from said longitudinal axis.

31. A cannula as set forth in claim 30 wherein the port of the needle is elongate in shape and has a minimum dimension of about 0.0155 in.

32. Vessels for placement in a series of vertical cylindric wells in a parallel reactor, said reactor having cannula passages extending at an angle off vertical from an exterior surface of the reactor to said wells, each cannula passage being adapted for the passage therethrough of a cannula containing reaction material to be delivered to a respective vessel, each vessel having a bottom and a cylindric side wall extending up from the bottom and terminating in a rim defining an open upper end of the vessel, said cylindric side wall having an inside diameter in the range of 0.5-2.5 in., said vessel having a volume in the range of 5-200 ml. and having an overall height in the range of 1.0-4.0 in. whereby when the vessel is placed in said well, the open upper end of the vessel is disposed at an elevation below said cannula passage where the cannula passage enters the well and is positioned for entry of the cannula down through the open upper end of the vessel to a position below said rim for the delivery of reactant materials into the vessel.

33. A method of preparing and delivering a slurry into a series of vessels in a reactor, said method comprising:

(1) mixing a particulate solid material and a liquid dispersing medium and agitating the mixture to form a substantially homogeneous first slurry in which said particulate solid material is suspended in the liquid;

(2) aspirating said first slurry into a cannula carried by a robot system while the slurry is substantially homogeneous,

(3) operating the robot system to insert the cannula into the reactor;

(4) delivering the slurry from the cannula into the vessel while the cannula is in said reactor, and

(5) repeating steps 2-4 for a second vessel and optionally a second slurry.

34. A method as set forth in claim 33 wherein said aspirating occurs during said agitating.

35. A method as set forth in claim 33 wherein said slurry is delivered to said vessel while the slurry is still substantially homogenous.

36. A method as set forth in claim 35 wherein said slurry is delivered to said vessel within 60 seconds of said aspirating.

37. A method as set forth in claim 33 wherein said agitating is accomplished by vortexing.

38. A method as set forth in claim 33 further comprising aspirating a barrier liquid into said cannula after aspirating said slurry and before delivering said slurry.

39. A method as set forth in claim 33 wherein said slurry comprises a catalyst.

40. A method as set forth in claim 39 wherein said catalyst is supported on said particulate solid material.

41. A method as set forth in claim 33 wherein said slurry is prepared less than 90 minutes before delivery to said first vessel.

42. A method as set forth in claim 33 wherein said vessel into which said slurry is delivered is pressurized.

43. A method as set forth in claim 33 wherein said first and second slurries are of different composition.